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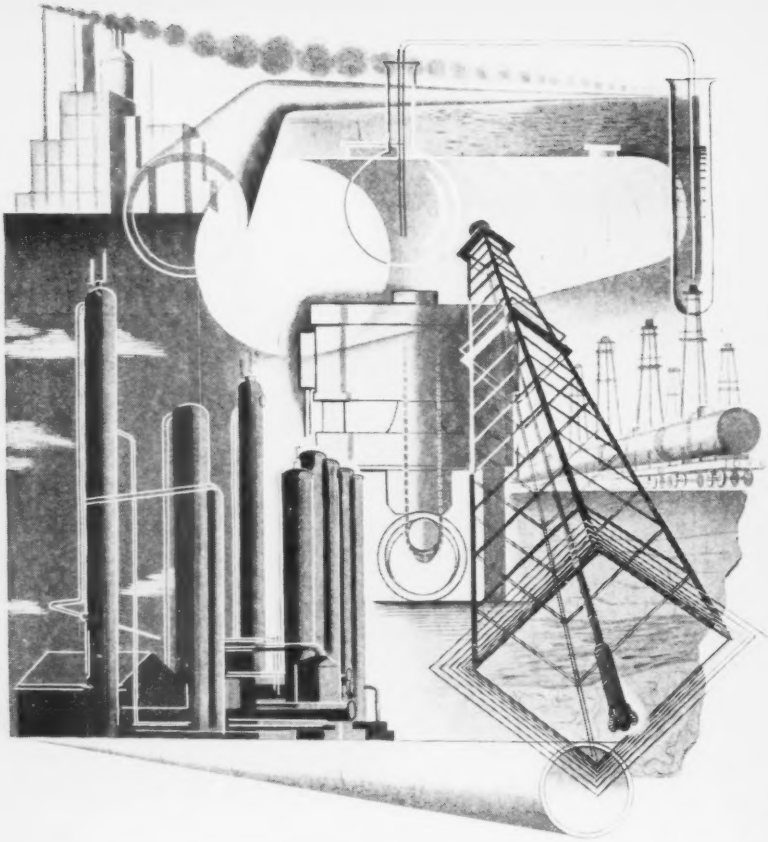

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NEW PHYSICAL METHODS IN THE PETROLEUM INDUSTRY*

By K. G. BRUMMAGE, P. W. COOKE AND R. R. GORDON†

ELECTRON DIFFRACTION

THE PRINCIPLES OF DIFFRACTION

THE methods of analysis described in the section on spectroscopy have utilized the principles of the refraction of radiation to obtain dispersion of various wavelengths into a spectrum. It is possible to achieve similar dispersion by the use of a diffraction grating, which consists, for optical wavelengths, of a set of closely and equally spaced lines on a piece of glass or metal.

The ability to undergo diffraction is common to all wave motions and is, indeed, a criterion of their wave nature. In the diffraction of monochromatic radiation the incident rays fall upon a row, or rows, of equally spaced scattering points, for example the lines on an optical grating, and each point becomes the centre of a system of radiating wavelets, each with the same amplitude and all possessing the wavelength of the main beam. The waves from the various centres become superimposed upon each other, and the result is to reinforce the scattered radiation in certain directions and to cancel it out in others, according as the superimposed waves are in or out of phase. Hence, the effect of diffraction is to give rise to secondary rays in directions different from the original, and dependent upon the spacing of the scattering points or lines and also upon the wavelength. The phenomenon can, therefore, be used either to sort out different wavelengths,

* Continued from page 49.

† Research Laboratories, Anglo-Iranian Oil Co., Ltd.

as in a diffraction grating spectrograph, or to provide information regarding the grating dimensions.

If a is the spacing between lines or scattering centres and λ is the wavelength of the radiation, the angle made by a diffracted beam with the row or grating is given by α_n in the expression

$$a(\cos\alpha_n - \cos\alpha_0) = n\lambda$$

where α_n is the angle between the row and the incident rays, and n is an integer (see Fig. 11). Thus, a and λ

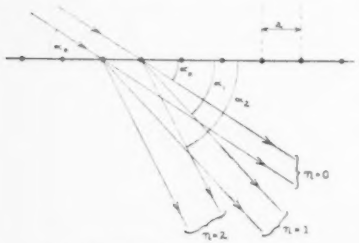


Fig. 11.—Diffraction by a grating

must be of similar magnitude for diffraction to be evident. It is, therefore, possible to obtain optical gratings by ruling on glass or metal; about 14,000 lines to the inch are required. With smaller wavelengths, such as occur in X-rays, much smaller gratings are necessary, and crystals are employed as three-dimensional gratings, with the regular distances between the atoms, ions or molecules acting as the diffracting spacings. X-ray diffraction with rays of known wavelength is therefore a powerful tool in the elucidation of crystal structures.

Similar diffraction effects have been obtained using electron beams instead of X-rays, and it it now firmly established that electrons in motion have waves associated with them, just as electromagnetic waves behave in some respects as particles. The wavelength of an electron beam which has been accelerated through a voltage V is given roughly by

$$\lambda = \sqrt{\frac{150}{V}} \text{ Ångströms.}$$

Thus a 50 kilovolt beam has a wavelength of about 0.05 Ångström, or 5×10^{-10} cm.

Application of Electron Diffraction to Lubrication

X-rays and electrons may both be used for the investigation of interatomic spacings and crystal lattices, but the two techinques differ in that electrons are scattered and absorbed by matter far more easily than X-rays. In consequence, while X-rays are useful for examining specimens in bulk, electrons can penetrate only to a limited extent, and give information applicable only to the surfaces; alternatively very thin films can be studied. It is the ability of electron diffraction to investigate the structures of surfaces and of films on surfaces that makes it a useful tool in lubrication research.

Thick or fluid film lubrication, where the oil film behaves as a liquid, so that the frictional characteristics can be calculated using hydrodynamic theory, is now well understood, but much remains to be learnt about boundary lubrication. Here the oil film is so thin that it no longer has the properties of a liquid; the load is taken by absorbed films of oil molecules on each bearing surface, and the surfaces may touch each other through the oil and thus give rise to wear. The friction observed under these conditions is not dependent upon oil viscosity; in fact, the mode of action of the oil is to a large extent unknown, and is described by such vague terms as "oiliness".

Under these conditions, the structure and orientation of the lubricant film is of great importance and the chemical composition, structure and finish of the surfaces also have their influence upon the friction and wear. In the examination both of the lubricating film and

of the surfaces, electron diffraction technique can play a useful role.

Work so far has been largely concerned with the lubricant film structure. Since lubricating oils are extremely complicated, the subject has been approached by studying pure hydrocarbons, acids and esters of high molecular weight, and finding the dependence of their structure in thin films upon rubbing and heating, and also the influence upon them of different substrate metals.

Apparatus and Technique

The electron diffraction camera employed was of the Finch type, and a drawing of it is shown in Fig. 12. It is continuously evacuated by the mercury diffusion pump shown on the right. In order to allow an electrical discharge in the chamber at the top of the camera, however, a slow leak of air flows into it through capillary copper tubing. The electron beam produced in the discharge chamber, the cathode of which is maintained at 50 kilovolts below earth potential, passes through a pin-hole in the anode diaphragm and travels down the camera, ultimately falling upon a horizontal zinc sulphide fluorescent screen in the camera block. The spot of light produced on the screen in this way is focused by the adjustable focusing coil, carrying a current of about 0.2 amp. The specimen is inserted through one of the ports, on a specimen carrier incorporating metal bellows, to permit adjustment of the specimen position. All vacuum joints are made using ground and greased metal surfaces.

The diffraction pattern produced on the fluorescent screen is recorded by turning the screen back on a shutter, thus exposing a photographic plate beneath it to the electrons. The plate holder at present in use carries three plates which may be successively exposed without breaking vacuum. For experiments upon the heating of organic films on metals, a special heated specimen holder has been designed, with an electrical heater beneath the surface under examination, and a thermocouple for temperature measurements. Surface blocks of various metals were available and were interchangeable.

The films to be investigated were deposited either on polished metal discs

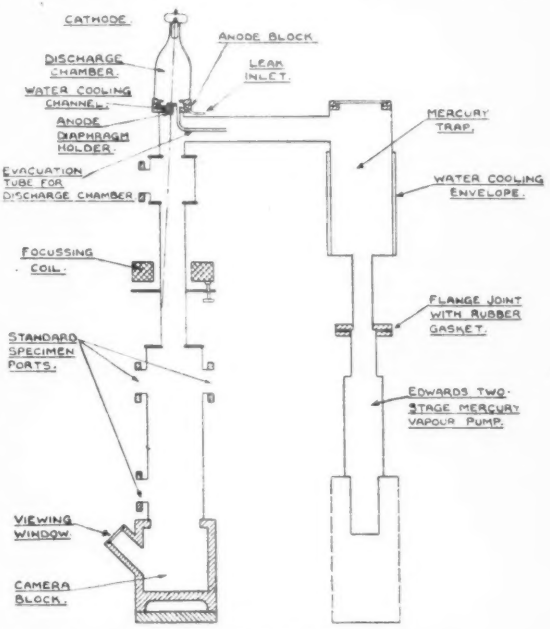


Fig. 12.—Diffraction Camera

or on thin collodion films. In the former case, the electron beam was arranged to fall at glancing incidence on the surface, to give a "reflection" pattern, and in the latter it passed normally through the film, yielding a "transmission" pattern. Deposition was generally effected by evaporating a known quantity of solution upon the surface, taking care to spread the layer evenly by rotating the specimen in an atmosphere nearly saturated with solvent vapour. In the case of polar materials, some films were obtained by forming a monolayer on water. The requisite number of layers was then built up by repeated dipping of the specimen disc beneath the surface of the water.

Experimental Results at Room Temperature—Unrubbed Films

The materials which have been examined include tetracosane ($n-C_{24}H_{50}$), triacontane ($n-C_{30}H_{62}$), tetratriacontane ($n-C_{34}H_{70}$), palmitic acid ($n-C_{15}H_{31}COOH$), stearic acid ($n-C_{17}H_{35}COOH$), behenic acid ($n-C_{21}H_{43}COOH$), methyl stearate ($C_{17}H_{35}COOCH_3$), ethyl stearate ($C_{17}H_{35}COOC_2H_5$) and cetyl palmitate ($C_{15}H_{31}COOC_{16}H_{33}$), deposited on stainless steel, copper or collodion.

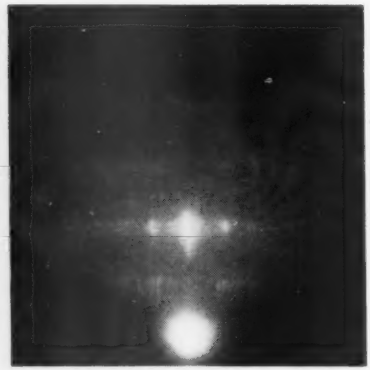


Fig. 13

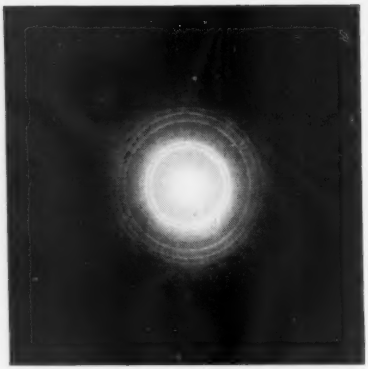


Fig. 14

The substrate exerted little influence upon the film structure, and the results were all of the same general nature, irrespective of the film composition. A typical reflection pattern from a normal paraffin on stainless steel is shown in Fig. 13, and Fig. 14 is the type of transmission pattern (beam normal to the film on collodion) obtained from all materials. The grouping of the diffractions of Fig. 13 on a set of equally spaced horizontal lines, together with the fact that the same pattern is obtained whatever the direction of the electron beam across the surface, may be interpreted as showing that the long axes of the molecular chains were normal to the surface, and were disposed randomly about the surface normal. Also, the occurrence of vertical streaks on the horizontal intensity zones is an indication that the molecules were grouped into crystals, and that these crystals were randomly arranged about the normal. The interpretation of fatty acid and ester photographs is very similar.

All the films on collodion gave by transmission the rings of Fig. 14. They are due to a random array of crystals with the long molecular axes parallel to the electron beam, and therefore normal to the collodion surface; the transmission results therefore confirm the reflection observations. The transmission

patterns from different materials were all similar because the cross-section of all the crystals normal to the molecular axes, and, therefore, to the incident electron beam, were similar, that is, the lateral packing of the paraffinic chains was always the same.

The Effect of Rubbing Paraffinic Films

If a thin film of a paraffin, a fatty acid, or an ester upon a metal was subjected to shear by rubbing it on, say, filter paper under manual pressure, the crystals were afterwards found to have taken up new orientations on the surface. The molecular chains were aligned in the direction of rubbing and making definite angles with the surface, determined by the crystal structure. Figs. 15 and 16 are typical photographs from a rubbed stearic acid film, Fig. 15 with the beam parallel to the rubbing direction and Fig. 16 with it at right angles to this direction. These two photographs show the most common orientation after rubbing, that is with the molecular axes making a small angle of about 5 degrees with the surface, this angle subtending the direction from which the paper rubbing surface approached, as shown in Fig. 17. The

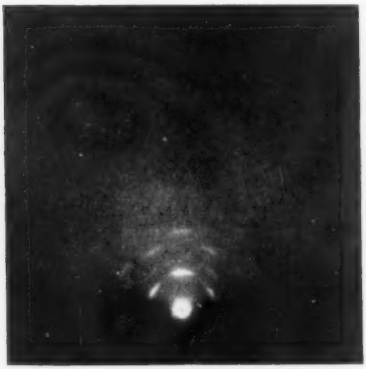


Fig. 15

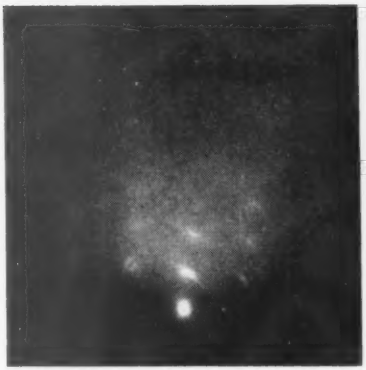


Fig. 16

molecules were never aligned so as to make a small angle with the surface while pointing in the opposite direction from that of Fig. 17, because the crystals were rigid, so that those lying as shown in Fig. 17 would be pressed on to the surface, whereas those aligned in the opposite direction would be pulled away by the passage of the rubbing surface.

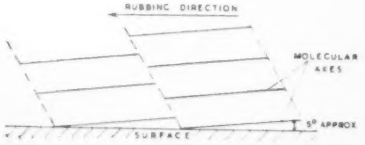


Fig. 17.—Alignment of paraffinic molecules after rubbing

It will be shown shortly that the fresh crystal orientations found after rubbing were due to re-crystallization of the film. The angles made by the chains with the surface are determined by the crystal structure, and they can be calculated using the general condition that the crystals must lie in such a manner that the plane substrate surface does not cut or intersect any of the molecular chains. The angles calculated in this way are in complete agreement with those observed.

The Heating of Films upon Metals

It is well known that in boundary lubrication the temperatures of the rubbing surfaces may rise considerably, and it is, therefore, necessary to examine the effect of heat on thin films. The heated specimen mentioned earlier was used for this purpose, and it was found that with all the materials studied the diffraction pattern gradually became diffuse as the temperature rose, and finally faded. On allowing the surface to cool, the pattern re-appeared, showing that the change was due not to evaporation of the film but to a breakdown of crystal structure to give what is known as an "expanded" film of individual molecules having one end attached to the substrate, but having the molecular axes distributed randomly in space. The temperature at which the screen apparently became blank was called the disorientation temperature, and it was found to be a function of the film composition and thickness and of the substrate metal. It was lowest for thin films and reached a constant value for films greater than about twenty molecules thick. Typical curves of disorientation temperature vs thickness are given in Fig. 18.

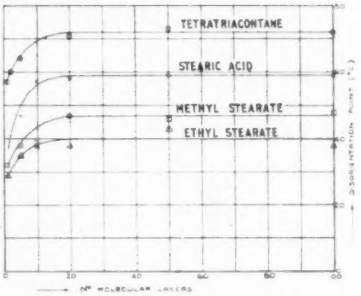


Fig. 18.—Disorientation points of films upon stainless steel

The thick-film disorientation temperatures for four materials upon a variety of metals are shown in Table III, together with the corresponding melting points.

Table III

THICK FILM DISORIENTATION POINTS (°C)

Metal	*Film Composition*			
	Tetratriacontane	*Stearic Acid*	*Methyl Stearate*	*Ethyl Stearate*
Aluminium	68	55	—	—
Cadmium	68	100	—	—
Copper	72	108	47	43
Nickel	69	95	55	44
Silver	74	100	—	44
Mild steel	74	105	44	56
Stainless steel	72	59	47	40
Melting-point	76	72	38	34

These temperatures are all comparatively low and suggest that the lubricant film in the majority of practical cases must be of the expanded type as defined above. The orientations which have been observed by electron diffraction after rubbing on filter paper are easily explained on the basis that the rubbing process caused expansion of the films and the expanded films subsequently recrystallized.

The disorientation temperatures of thick films of paraffins are always a few degrees below their melting-points, and those of esters with short "alcohol" chains are always above. Cetyl palmitate, however behaves in the same way as a normal paraffin. The disorientation points of thick films of fatty acids on different metals vary widely. In the cases of cadmium, copper and mild steel, the high values are due to the formation of metal stearates. The other metals may be divided into two groups, in one of which (aluminium and stainless steel) the acids behave in the same way as paraffins and become disorientated below their melting-points, and in the other (nickel and silver), they are similar to esters and become expanded above their melting-points.

A considerable degree of correlation has been observed between disorientation temperatures and the temperatures of friction discontinuties found using Bowden-Leben type friction apparatus. It demonstrates the effect of expansion of the lubricant film upon friction, and also makes it clear that the expanded film is an effective lubricant, because the break-down of lubrication is not observed until temperatures much higher than that of disorientation are reached.

It is evident that electron diffraction technique is a useful tool in the fundamental study of boundary lubrication. It has made the sequence of events in rubbing a thin organic film much clearer than has hitherto been possible. Attention is now being directed chiefly upon the functions of the bearing surfaces in boundary lubrication, and upon the changes brought about in them by running them together in the presence of a lubricant.

X-RAY DIFFRACTION

The ability of X-rays to undergo diffraction by crystals, and their high penetration into matter have both been extensively utilized in industry. Radiography, which depends upon their penetrating power, is not greatly used in petroleum research; it depends upon the relative adsorption by different materials of the X-rays employed. X-ray diffraction, however, is becoming increasingly important in the determination of chemical composition, phase transformations, crystal size and growth, size distribution, crystal orientation and crystal structure.

The Production of X-Rays

X-rays are formed when high-speed electrons bombard matter. The electrons are usually emitted from an incandescent filament in vacuo, and are accelerated by an electric field. They fall upon a metal target, and X-rays are produced at this target. The radiation from a particular target is of two kinds, the general or "white" radiation, and the characteristic radiation, determined by the target metal.

The "white" radiation consists of a continuous spectrum of wavelengths, with a sharp lower wavelength limit corresponding to the maximum energy of the incident electrons, and independent of the target material. This cut-off wavelength decreases as the accelerating voltage of the electrons becomes greater. The intensity of the general radiation rises to a maximum at a wavelength approximately 1.3 times the shortest, and then falls off gradually with increasing wavelength. The mechanism of the production of this "white radiation" has not been satisfactorily explained.

The characteristic radiation is super-

imposed on the continuous spectrum, and consists of several series of monochromatic radiations. It is formed as a result of collisions between the bombarding electrons and metal atoms in the target. These collisions cause planetary electrons to be expelled from the atoms. Subsequently, electrons at higher energy levels replace them, losing their additional energy in the form of radiation of wavelengths varying from a fraction of an Ångström (10^{-8} cm) to several hundred Ångströms. Since the number of permissible electronic energy levels is very small, the number of wavelengths is small, and they are grouped into series depending upon the energy levels to which the electrons fall during the emission of radiation. These series are designated K, L, M, N. . . . depending upon the lower energy level involved. The wavelengths observed are, therefore, dependent upon the energy levels in the target atoms, and also depend upon their atomic number, so that they are determined by the target material.

The simplest series of radiations from a given target is the K series, containing four principle wavelengths, the γ (a close doublet), the β (also a close doublet) and the doublet α_1 and α_2, in order of increasing wavelength. The δ and ζ components may be removed by suitable filters, leaving the α doublet. Resolution of this into α_1 and α_2 requires the use of a monochromator. For most diffraction work, the α doublet is used.

Common target materials are copper, cobalt, iron and molybdenum. A high melting-point is necessary because most of the energy of the bombarding electrons is dissipated as heat in the target. For this reason, the targets are generally water-cooled, and in the case of metals of low heat conductivity, the targets are made by electrodeposition on copper.

Apparatus

The apparatus in use at Sunbury is the Metropolitan Vickers Raymax

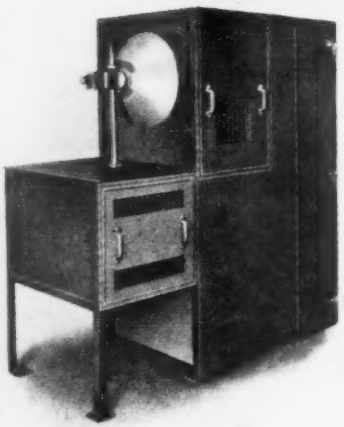


Fig. 19.—Metropolitan-Vickers Crystallographic X-Ray Unit

Industrial X-Ray Unit shown in Figs. 19 and 20. The demountable tube is continuously evacuated to a pressure of 10^{-5} mm. of mercury. The pumping and high tension equipment is mounted inside the cabinet. The whole sequence of pumping and high voltage production is relayed and interlocked to make the operation safe and foolproof. The X-rays emerge from two aluminium windows, one on each side of the tube and it is thus possible to use two X-ray cameras simultaneously.

An X-ray camera is essentially a piece of equipment designed to collimate the X-ray beam from the tube on to a specimen, mounted on a carrier which can be rotated, oscillated, or set at any inclination to the incident beam. Integral with the collimator and specimen holder is a film holder to record any diffraction pattern arising from the interaction of the beam and the specimen. The cameras in existence vary in type, each being designed to accommodate a specimen of a particular type, such as a single crystal, a crystal aggregate, a thin film, or a large object.

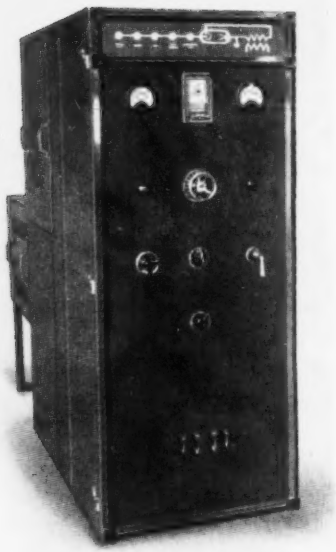


Fig. 20.—Control Panel

X-ray Diffraction Patterns

The pattern recorded on the film of an X-ray camera constitutes the diffraction pattern of the object under examination. The geometry of this diffraction pattern is determined by the spacings between the atoms or molecules in the object, and by the design of the camera. The intensity of each diffraction spot or line depends upon the types and arrangement of the atoms present, and it is possible to calculate the complete structure if sufficient diffraction data are available. This is the basis of crystal structure determination. Such determinations are, however, lengthy procedures and are rarely carried out in industry, which is concerned not so much with final structure determination, as with the relationship between various physical properties of a material and its diffraction pattern. Any change in the final arrangement of the atoms or groups of atoms in the material affects the intensities of the diffractions, their positions, or both.

In industry, including the oil industry, X-rays have been applied to the following fields:—

(*a*) Chemical analysis—both qualitative and quantitative
(*b*) Studies of transitions
(*c*) Identification of materials
(*d*) Crystal size determinations, size distribution, crystal growth
(*e*) Lubrication
(*f*) Examination of wear products
(*g*) Structure of petroleum hydrocarbons

(*a*) Chemical Analysis

Since a diffraction pattern is related not only to the repetitional distances in the crystal, but also to the number and types of atoms composing the crystal. then each chemically distinct compound will give a unique diffraction pattern. Figs. 21 (*a*) and (*b*) show the powder patterns produced by α-alumina (Al_2O_3) and by Boehmite (a hydrated aluminium oxide, $Al_2O_3.H_2O$). The pattern given by a mixture will contain the patterns due to each component. Fig. 21 (*c*) shows the pattern yielded by a mixture of α-Al_2O_3 and Boehmite. For a rough qualitative analysis only the positions of the diffraction lines are required with their approximate relative intensities, for comparison with the A.S.T.M. card index of pure compounds now being built up.

For quantitative analysis, accurate intensity measurements are required together with some internal standard method, whereby a known amount of a standard material is mixed with the specimen before introduction into the camera. A comparison of the intensities of the known and unknown lines can then be used to determine accurately the relative proportions of the constituents, in a manner similar to that used in emission spectroscopy.

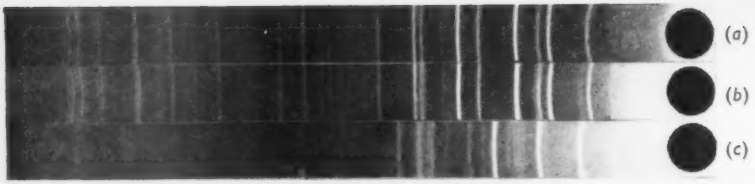


Fig. 21.—Analysis of Mixture
(a) α-alumina (b) Mixture (c) Boehmite

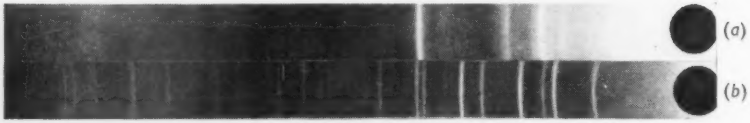


Fig. 22.—Allotropes
(a) γ-alumina (b) α-alumina

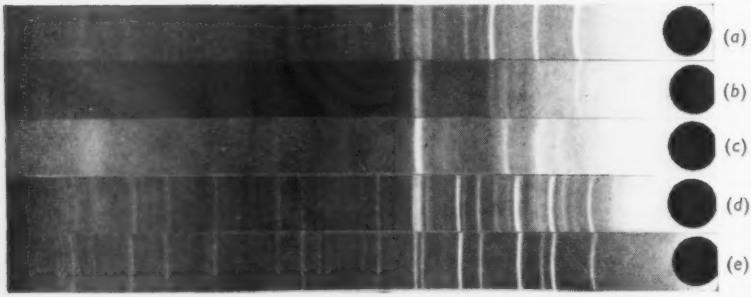


Fig. 23.—Transitions from Boehmite to α-alumina
(a) Boehmite γ-alumina plus Boehmite (c) γ-alumina
(d) α plus kappa alumina (e) α-alumina

Fig. 24.—Identification of Materials
(a) Residue ex plant (b) Ammonium sulphate

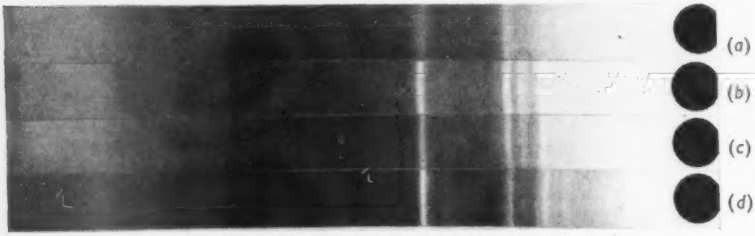


Fig. 25.—Crystal growth of γ-alumina (sub-microscope size)
(a) 30A crystal size (b) 50A crystal size (c) 70A crystal size (d) 100A crystal size

Pure chemical analysis is incapable of distinguishing between allotropes, and it is in this field that X-rays are indispensable. It is well known that alumina is widely used as a catalyst and a support for other active materials. It exists in a large number of different forms, both hydrated and dehydrated. The most active anhydrous form is the γ variety, while α-alumina is the stable relatively inactive modification. X-ray patterns of the γ and α varieties are shown in Fig. 22. They are structurally different, although indistinguishable chemically.

(*b*) Phase Transition Studies

Transition temperatures can be studied by X-rays used in conjunction with heat treatment. Specimens of one allotrope preheated for varying times at various temperatures yield patterns indicative of the degree of transition, and indicate whether the transition is sharp or otherwise. Fig. 23 shows the transition of Boehmite to γ-alumina and thence to α-alumina. A more modern development is the use of a high temperature X-ray camera, in which the specimen is surrounded by a furnace and may be maintained at any desired temperature whilst a pattern is being taken. This should obviate any erroneous conclusions that might have been drawn from examining a material at room temperature and assuming that its physical state was then the same as at the temperature at which it was used in the plant. The transition rate from γ-Al_2O_3 to the α-form is negligible below 1,000°C and is only complete after several hours at 1,100°C at atmospheric pressure. X-rays were instrumental in showing that in the presence of steam, this transition rate can be greatly accelerated and can even be completed at a temperature as low as 460°C after 14 hours treatment.[6]

(*c*) Identification of Materials

X-rays give a quick means of identifying materials. Only a minute quantity of material is necessary (less than a milligram) and the pattern obtained can be unequivocably matched with a set of standards. An example is shown in Fig. 24, where material removed from the standpipe of a catalytic cracking plant was found to be ammonium sulphate.

(*d*) Determination of Crystal Size, Crystal Growth, and Crystal Size Distribution

As the crystal size of a material decreases, there are fewer repetitions of the unit cell in the crystals and the X-ray spectra become progressively more diffuse. (The optical analogy here, is the ruling of fewer lines on an optical grating, leading to diffuse spectra). Various formulae have been derived correlating the average crystal size of the material with the measured width of the diffraction line at a particular fraction of its maximum intensity. Materials yield sharp diffraction lines when the average crystal size is between 5×10^{-4} cm and 2×10^{-5} cm (50,000–2000 Å or $5.0-0.2\mu$). As the crystal size falls below 2000 Å, the diffraction lines broaden. Fig. 25 shows a series of γ-aluminas of various crystal sizes, together with the heat treatment producing the sizes. Since catalytic activity is essentially a function of available surface area (on which the reactions take place), and this is a function of crystal size, the importance of size determination is obvious in catalyst studies. Sizes down to 10Å can be determined by this method. The best and easiest determination of crystal size is by use of the Geiger counter instead of by photographic recording of the diffuse bands, with subsequent microphotometry. Crystal growth can obviously be studied by this method.

Above 5×10^{-4} cm, the nature of the continuous clearly defined diffraction lines changes to a "spotty" type of pattern as the size increases, and it is

possible to determine crystal sizes in the region 5×10^{-4} cm to 10^{-2} cm by X-rays. Fig. 26 shows two samples of quartz of approximately 50—75μ and 5—20μ average crystal size.

In dealing with catalyst particles, each particle is usually composed of a number of crystals, and the size of the complete aggregate is of considerable interest. For particles with sizes up ot about 1000Å, the aggregates themselves act as scattering centres, owing to the difference of their refractive index (for X-rays) from that of the surrounding medium (usually air or a vacuum). The distance, a, in the general diffraction equation

$$a(\cos\alpha_n - \cos\alpha_0) = n\ \lambda$$

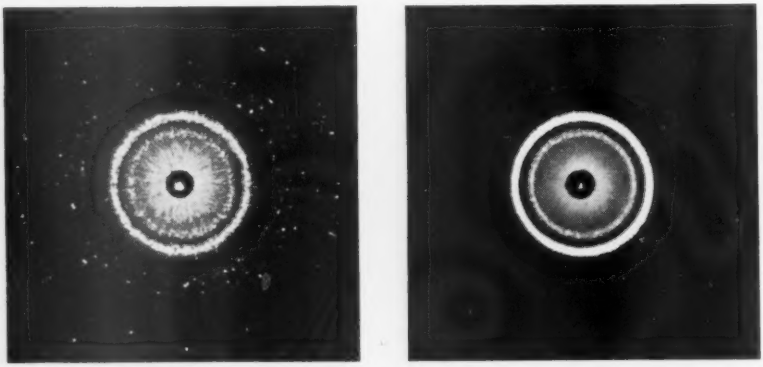
is, therefore, of the order of 1000Å, and the deviation of the beam $(\alpha_n - \alpha_0)$ is only of the order of minutes of arc. In order to record these low angle diffractions, a large camera radius is required, and the primary beam must be absorbed to prevent obliteration of the lines. This is the basis of the small angle scattering technique originally used to determine the large spacings in proteins and fibres (100—1000)Å. The method has been extended to the determination of particle sizes of material in the range 0—1000Å, and to the derivation of particle size distributions in this range.

(*e*) Lubrication Studies

Although no X-ray work in this field has been carried out at Sunbury, much information has been obtained in other laboratories (notably in America), on lubricating films of the "boundary" type. X-ray analyses of lubricating films of oils and greases have fully confirmed the oriented film layers. The most comprehensive X-ray studies of lubricants have been made by Clark, Sterrett and Lincoln.[7]

(*f*) Examination of Wear Products[8]

Powders produced by the abrasion of steels in a wear testing machine have been examined by X-rays. The abrasion products consisted of mixtures of the parent metal (ferrite) and iron oxide. The oxides observed were FeO and Fe_3O_4, but never Fe_2O_3. The relative amounts of α-Fe, FeO and Fe_3O_4 in the wear powder depended characteristically on the composition of the steel, its phase constitution and other factors.



50-75μ Quartz *5-20μ Quartz*

Fig. 26.—Crystal size in the microscope range

(*g*) Structure of Petroleum Hydrocarbons

A wealth of information about the structure of long chain hydrocarbons, cycloparaffins and ordinary paraffin wax has been obtained by many workers, notably Muller, Shearer, and Clark. The difficulties involved are enormous since petroleum consists of a very large number of different hydrocarbons, and such difficulties are evident when the study of ordinary paraffin waxes is attempted. Clark[9] has shown that in spite of the fact that these waxes may contain as many as 18-20 different hydrocarbons, both normal and branched chain isomers, a single diffraction spacing corresponding to molecular length is obtained, together with the usual side spacings. Thus, analysis of waxes by X-rays is almost impossible, but some attempt has been made to correlate the molecular weights (as determined by X-rays) with those determined by other methods, and information on molecular form, polymorphism, molecular weight and isomerism, has been classified.

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A B.I.O.S. TRIP TO GERMANY, 1946*

By L. IVANOVSZKY

In the following paper is given a description of the actual working of a B.I.O.S. (British Intelligence Objectives Sub-committee) technical investigation team, the functions and aims of the B.I.O.S. organization and of F.I.A.T. (Field Intelligence Agency Technical), (its U.S. counterpart), together with some unbiased remarks concerning the present situation in Germany.

For various reasons, little has become known in the past concerning the B.I.O.S. organization and its American twin-sister, F.I.A.T. It seems that the authorities had become aware of this lack of knowledge, hence the B.I.O.S. exhibition opened in London by Sir Stafford Cripps on December 9th. The U.S. authorities have already given adequate recognition to the F.I.A.T. organization.

Before proceeding further, I shall endeavour to trace the interesting history of the B.I.O.S.–F.I.A.T. organizations, which originated from the Technical Information Section of S.H.A.E.F., a body chiefly interested in intelligence on materials of war.

When Rome fell, a small force of specially selected and trained technicians came into being for the first time.

They were so successful that in the summer of 1944 the T-Forces were organized by the Combined (U.K. and U.S.) Intelligence Objectives Sub-committees known as C.I.O.S. They moved with spearheads and even in advance of armies to seize enemy equipment and records before these could be destroyed by the enemy or by misdirected attempts at revenge by labourers who were "displaced persons"; valuable materials and vital records were sent back safely to the rear. Later, a continuous stream of combined British and American teams of technical investigators was sent to the Continent to assess and study industrial and

*Condensed from a paper read before Northern Branch, Institute of Petroleum, December 17, 1946.

scientific targets in Germany and formerly occupied countries and to screen and collect documents.

With the end of the war, the drive for industrial and scientific information began to outweigh military considerations, and after the dissolution of S.H.A.E.F., C.I.O.S. ceased to exist in July 1945. Its work is carried on by B.I.O.S. in the U.K. and F.I.A.T. in the U.S. Both these organizations continue to work in close co-operation not only for the mutual benefit of both countries, but also for that of all non-enemy nations. The name T-Force is no longer used within the U.S. organization, but there is a military body called T-Force which is an organ of the Control Commission for Germany, British Element. "T" stands for Target. The functions of T-Force will be explained later.

ORGANIZATION OF B.I.O.S.–FIAT

Requests for information from industry and scientific institutions are received by the appropriate ministry and studied by its planning section. After consultation with representatives of the industry concerned and the co-operation of experts, a study group is formed if there seems a need for information. The members of the team to be sent to Germany are elected by a meeting representing the industry, and in consequence, target lists are prepared. In close consultation with the Technical Intelligence Section of B.I.O.S., London, their liaison officers in Germany and the respective sections of the Control Commission for Germany, consideration is given to the desirability or otherwise of visiting the various targets proposed. The Movement Section of B.I.O.S. then arranges the transport of the team, and the final step before its ultimate departure comprises technical and administrative briefing.

Upon arrival in the British zone in Germany, the team is messed and billeted by the T-Forces organization, which is also responsible for the transport within the zone and the allocation of cars to the team (normally there is one Humber for three investigators). The main T-Forces Messes (receiving camps) are in Herford and Löhne; there are, in the zone, ten more messes and the Reichshof Hotel in Hamburg.

The team reports first to the B.I.O.S. administrative officer, B.I.O.S. Office, H.Q. T-Force, at Bad Oeynhausen, where it is again briefed. After reporting to the Military Government at Minden and consulting the particular sections of its Trade and Industry Division, clearance has to be obtained from the corresponding branches of the Regional and Local Military Governments (e.g. the North German Oil Control). All German industrial establishments in the British zone are controlled through five Regional Military Government Headquarters. These formalities having been concluded, the targets can then be visited.

Brief summaries of the outstanding facts uncovered during the visit to each individual target have to be submitted before the team leaves the zone. They are known as "Interim Reports" and copies are forwarded to the B.I.O.S. Secretariat.

The "Field Team Report," as the final report is named, has to be prepared as soon as possible after return to the U.K. and then submitted to the B.I.O.S. Technical Intelligence Section which forwards it to the sponsoring ministry for "vetting" and allocation of security classification. It is then ready for publication by His Majesty's Stationery Office.

The procedure in the two other zones follows roughly the same pattern. At F.I.A.T. Main, Hoechst, British F.I.A.T. directs the work of British teams visiting targets in the U.S. and French zones. At Gengenbach, seat of the French

F.I.A.T., the "Section d'Information Scientifique" has a British Liaison Officer. In the U.S. zone, the "Trade and Commerce Section" is the body corresponding to the "Trade and Industry Division" in the British zone.

In Berlin there is a British organization called F.I.A.T. (Forward) and a U.S. F.I.A.T. office.

In the U.S. and French zones, British Investigators are messed and billeted in institutions run by the respective Military Governments. In Hoechst, British Investigators are billeted in three small houses run by T-Force. Transport is provided by a small British military detachment affiliated to T-Force.

B.I.O.S.–F.I.A.T. ACHIEVEMENTS

After this brief introduction, it is perhaps appropriate briefly to deal with the achievements of the B.I.O.S.–F.I.A.T. organizations. B.I.O.S., it should be explained, is an inter-departmental body under the sponsorship of the Board of Trade. The latter also runs the "Foreign Document Unit" at Landsdowne House. This unit is the main central repository for the great number of original German documents in our possession. It provides facilities for translating, abstracting, and supplying copies to any interested party.

In the U.S., the Department of Commerce, and its elements the Office of the Publication Board and the Office of Technical Service, direct the work of U.S. F.I.A.T.

So far, more than 10,000 investigators, industrialists, technicians, scientists, and so on have gone to Germany in 3000 separate teams and have visited more than 10,000 separate targets—factories, scientific institutions, business premises, and many other objectives, amongst them about 1000 fuel and power and chemical targets. The number of reports published already amounts to some 1400; many more will be issued before the task is completed. To the total number of reports, joint British–American teams have contributed 540, British teams 572, and American teams 278. 490,000 copies of final reports have been sold by H.M.S.O. to individuals and 460,000 distributed gratis to libraries, trade and research associations.

In the British zone the T-Force staff consists of some 2000 persons, and the British F.I.A.T. staff in the U.S. zone of about 250. In both zones, 700 vehicles are in service and have covered more than 13 million miles during the past year.

I assume that some information regarding the U.S. F.I.A.T. organization in Hoechst will be welcome. Some 1000 persons are employed, amongst them about 650 Germans and 20 Allied civilians and displaced persons. F.I.A.T.'s microfilm unit, probably the largest in the world, reads, translates, and classifies 30,000 pages of documents and blue-prints daily. By June next, when F.I.A.T. expects to close its operations, 7 million pages of records will have been microfilmed. It is estimated that the records of about 1600 targets will have to be screened; 5 per cent only are thought to be worthy of microfilming. Since V.E. Day, 9000 investigators have made 5000 trips, in the course of which they have interviewed 100,000 Germans scientists and technicians and prepared 28,000 reports.

There seems to be some discrepancy between the number of trips and reports announced by the B.I.O.S. and F.I.A.T. organizations. The latter probably refers not to the final reports, but to the Interim Reports (although B.I.O.S. lists only 10,608 of that kind) and some of the teams may have undertaken more than one "trip." I believe that U.S. F.I.A.T. counts it as two trips if a team, which had worked in the U.S. zone, returns from another zone to visit additional targets. At all events, the figures referring to F.I.A.T. were recently

published in a U.S. Army paper *The Stars and Stripes Week-end.*

THE WAX TEAM

The team of which the writer was a member was sponsored and organized by the Ministry of Fuel and Power, and had the task of investigating into research, production, refining, and utilization of waxes and wax-like materials from all sources. It consisted of nine members, five being drawn from oil companies, three from wax refiners and blenders, and the team leader from the Fuel Research Station. In order to visit a large number of targets in a comparatively short time, two, or sometimes three, sub-teams visited different targets simultaneously.

Having concluded its work in the Hamburg area, the team divided, six members going to the Ruhr. Of this number, two were sent to Berlin for a few days, the whole six returning to this country after an absence of about four weeks. The remaining three members of the team, myself amongst them, turned their attention to the U.S. and French zones. One of the three members had, unfortunately, to return to this country after about five weeks owing to ill-health, the other two returning after six and seven weeks respectively. On an average, therefore, the team spent about five weeks in Germany. Some 80 industrial, commercial, research and personal targets were visited, involving the interrogation of about 200 Germans. The time spent on any one target varied from a few hours to a whole day, in exceptional cases lasting even two days.

The interrogation of German personnel takes the form of a friendly discussion on specific technical questions, followed by an inspection of the works, institute or premises. As a whole, the work is both interesting and instructive, although the language difficulty and the reluctance of some of the Germans to answer questions freely and completely, sometimes makes the work rather tedious.

Amongst the targets visited were oil-shale works, petroleum refineries, wax refining and blending works, synthetic wax and synthetic vaseline plants, synthetic fatty acid works, research laboratories and a high-pressure research station, an oil mill de-oiling and fractionating paraffin gatch and petrolatum, a fish-meal plant extracting montan wax, emulsifying agent manufacturers, wax merchants, and a large plant specially designed to convert high-melting-point Fischer-Tropsch waxes, as well as petrolatums, paraffin gatch and the like into a range of waxes of more specific properties, thus conferring upon them a higher commercial and technical value. On the wax-using side, polish manufacturers, candle makers, waxed paper mills, cable works, rubber manufacturers, and others were visited.

LIFE IN GERMANY

Now for a few words concerning the "off-duty" life of investigators. In the British zone they are billeted in T-Force messes or hotels run by Germans under T-Force supervision. In most cases, accommodation is comfortable, and the meals substantial and well prepared. Investigators' leisure hours are not abundant, because after returning from a target, usually towards the late afternoon, the team assembles to exchange views, to discuss itinerary and working procedure for the next day, and to plan future work.

In the U.S. zone—at Hoechst to be precise—there are three small private houses which serve as British investigators' billets; they are not quite up to the standard of the accommodation in the British zone. In the other towns, British investigators are messed and billeted in U.S. officers' transit hotels; the food is plentiful and of good quality.

You will surely wish to learn something of the everyday life of a German. Well, all in all it is pretty grim. It must first of all be realized that most of the important industrial towns have been destroyed to an extent surpassing imagination.

Public services, however, have largely resumed normal working, tram-cars and underground trains are running, gas and electricity works are operating, and postal and telephone services have been restored. The tram-cars are abominably overcrowded, passengers hanging at the entrances like clusters of bees.

The Control Commission for Germany does its best to rehabilitate German industry and to restore the life of the nation, in co-operation with the newly created German Economic Office, the *Landes Wirtschaft Amt*, and in accordance with the Berlin Protocols. All plants which were in a condition to resume work or were capable of being easily repaired have been given permits and are supplied with material as far as it is available, if they fall within the categories of industry allowed by the Berlin Protocols and are necessary for German life. In addition, some forbidden industry has been temporarily permitted to alleviate the present German economic situation, such as the production of synthetic hydrocarbons for conversion into fatty acids, fats, soaps, etc.

It seems that there is work enough for all persons willing to co-operate. German police, organized and directed by the Control Commission's branches, and I may say apparently very well organized and directed, keep a close check on their countrymen, who must possess a kind of identity card to show that they are doing some legitimate work.

Hanover seems to be even more heavily damaged than Hamburg, and this is certainly true regarding Kiel, which is, perhaps, 80 per cent destroyed, or even more. I have heard, however, that Cologne and many places in the Ruhr have fared even worse. On the other hand, there are many country towns which have suffered little or no damage.

In Kiel, as in many other devastated areas, one sees overcrowded tram-cars and, as theatres, cinemas, shows, and other places of public entertainment are just as overcrowded, one begins to wonder whence those masses come and whither they go, as one rarely sees a house. I understand that they live largely in dug-outs, air-raid shelters, and in what is left of basements.

As Germans are not admitted to British messes, hotels, clubs, and most places of British sponsored entertainment and, *vice versa*, British personnel is barred from visiting most German places, fraternization is at least to some extent restricted. In order not to curtail German rations, British personnel is not allowed to have meals in German restaurants.

Comparing life and conditions in the three zones, I could distinguish very little difference.

Frankfurt, the seat of the U.S. Army H.Q., is probably more than 50 per cent destroyed. There are numerous attractive clubs for U.S. Army personnel and civilian employees in Frankfurt itself and in the health spas in the vicinity.

Hoechst, the headquarters of the F.I.A.T. organization, is amongst the lucky towns which escaped damage, together with the I.G. works. The I.G. works at Griesheim are partly destroyed. The extremely valuable library of I.G. documents now housed at the Griesheim works was one of our B.I.O.S. targets. Mannheim, Karlsruhe, Darmstadt, and Offenbach are a few more examples of heavily blitzed towns. A pleasant surprise, however, awaits one at Heidelberg, Germany's student town No. 1; outwardly, it still retains its pre-war appearance. The life of the university is in full swing, the population seems

more lively than in other places, and even the shop windows look more colourful than elsewhere.

In the French zone, Baden-Baden, the seat of the French Military Government H.Q., seems to be unharmed, but Ludwigshafen exhibits considerable damage, both the town and I.G. works being largely destroyed. Nevertheless, numerous plants have resumed production, and others might be restored without great difficulty. Mayence, too, has suffered heavy damage.

It should be borne in mind that the conditions of misery in Germany to-day are the direct outcome of that country's own actions. The destruction is so enormous and widespread that the tasks of reconstruction and rehabilitation constitute one of the most gigantic problems ever to confront mankind. I say mankind intentionally, because if the Allies are bound to help Germany for humane and common-sense reasons, is it not the obvious duty of all the other countries of the world which suffered neither losses nor hardships, but made handsome profits, to contribute to the heavy burden of Germany's reconstruction?

An essential pre-requisite is that Germany shall be purged of her Nazis and from all open or camouflaged Nazi ideology, both for her own benefit and for the sake of the future peace and prosperity of the world.

GERMAN PUBLICATIONS AND TECHNICAL DEVELOPMENTS

To end my talk I want to refer very briefly to a few interesting facts connected with the publication of papers and periodicals in Germany, and then to proceed to a review of some interesting technical developments which I encountered there.

The number of German bi-weeklies published under the supervision of the various Information Control Units is a fairly high one. The Military Authorities also produce several journals and bulletins in English, probably also in French and Russian. One of them, *The British Zone Review*, published by the British Element of the Control Commission for Germany (C.C.G., B.E.), and printed by the Printing and Stationery Service B.A.O.R., is an interesting fortnightly review of the activities of the C.C.G., B.E., and often contains articles of technical interest.

About 45 scientific and technical periodicals are being published in the U.S., French and Russian Zones, amongst them many newcomers. As far as I could ascertain, no periodicals of that kind are produced in the British zone. I was told that only 4 per cent of the German sulphate and sulphite production takes place in the British zone, and that consequently it was necessary to import paper from the U.K. even to maintain the public press at its present level. I understand that most of the German journals and periodicals are allowed only a very restricted circulation, chiefly owing to the scarcity of labour, paper, power, etc. It seems that some technical books are being published or republished in small editions, in addition to textbooks for schools, etc. Several publishers and firms concerned with the distribution of books have re-established their businesses in Frankfurt, having previously been extant in Leipzig.

I would now like to comment upon some of the more interesting technical points I encountered during my stay in Germany.

The use of aluminium chloride as a refining agent for hydrocarbon waxes has become common practice in Germany, particularly for those types which contain a large proportion of tar-forming compounds that gell when reacted with sulphuric acid at raised temperatures.

The so-called "Frederking vessels" which are widely used in Germany—

and I assume also in the U.S.A.—seem to be comparatively unknown in this country. These vessels are constructed of cast-iron, bronze, lead, or any other suitable metal, with coils of high-pressure tested stainless steel tubing cast in the walls. They can be made to any design to suit particular requirements, and can be equipped with several independent coils. The apparatus offers special advantages for operations involving either alternate heating and cooling or the use of gaseous or liquid heating media circulating through the coils under high pressures. The cast-in steel tubing, taking up the entire pressure of the heating medium, permits the walls to be made of metals such as lead, which are not suitable to withstand pressure. As walls and cast-in steel tubing form a uniform body, the efficacy of heat transfer is excellent, and the heating system is superior to any other, such as jackets or coils situated inside the vessel. Furthermore, local overheating becomes impossible and stirring, discharging and cleaning operations are not hampered by any obstructing objects inside the vessel. The application of Frederking vessels covers practically all branches and operations of chemical and processing industries such as distillation, evaporation, crystallization, drying, calcining, melting, refining, chemical treatment, etc. They can be built as trays, open or closed vessels, autoclaves, or other forms of vessel.

The band-filter principle has found in Germany some further applications and modifications. I have been told that Riebbeck A.G. has introduced a similar plant to extract montan wax from the coal-dust separated from the dried brown coal during the screening stage.

A modification of the same idea uses instead the moving shallow trays of the band-filter an endless metal-gauze band which is coated with a thin layer of a waxy material, before passing through one or more vessels containing solvents, in order to separate the material into various cuts by fractioned extraction.

In the extraction of montan wax from brown coal, I have been informed that the intermittent Soxhlet principle has been partly superseded by pump circulation of the solvent under a slight pressure.

In the British zone, montan wax is extracted from Helmstedt brown coal in a fish-meal works near Hamburg, making use of the partly idling extraction plant for this purpose.

The production of synthetic vaseline and ozokerite-like bodies by ketonization and subsequent hydrogenation of fatty acids from fish oils and of the distillation residue from synthetic fatty acids seems to open up many new possibilities.

Cake-moulding presses can be used only for non-tacky waxes; for the tacky variety a kind of slow-moving cooling elevator has been developed.

I have also heard of another plant characterized by a huge horizontal conveyor belt which transports the trays filled with molten paraffin wax through a cooling chamber.

Solvent-extraction filter presses with fully enclosed filtering devices may be useful implements to solve specific problems.

Many attempts were made to improve the usefulness of high-melting-point Fischer-Tropsch waxes. A successful solution has been found in fractionated solvent-extraction processes and blending some of the fractions with specially prepared cuts from petroleum products. High vacuum distillation processes were also used for this purpose.

The process of cold extraction found also application in the shale-oil industry to de-oil the yellow paraffin wax. The wax is atomised, the fine wax-powder mixed with cold benzine and the oil-containing solution separated by centrifuge. The wax is then twice washed in the centrifuge with fresh solvent.

Soaps are cut back in Germany up to 50 per cent with a urea-formaldehyde resin low-polymer. The latter probably forms a higher polymer during the boiling process. The U.F. resin, which is cheaper in Germany than the fatty acids which are also short in supply, offers many advantages as compared with the clay, formerly used for the filling of cheap soap.

Paraflow, which was introduced into the petroleum industry to depress crystallization of wax in lubricating oils, is added in very small quantities to the wax distillate of the T.T.H.* product. The idea is to prevent the formation of large crystals or crystal aggregations which include oil.

A clay has been found in Germany which yields a wax apparently very similar to montan wax.

The addition of Oppanol (poly*iso*-butylene or Vistanex) to paraffin wax improves the vapour-proof properties of paper coated with that mixture in comparison with paraffin-wax-coated paper.

* Tief Temperatur Hydrierung (low temperature hydrogenation).

LUBRICATION AND THE MOTOR CAR

By KENNETH C. HUNT, B.Sc., F.Inst.Pet.*

PERHAPS the most outstanding feature of the instruction manual which is given away with every new motor car is the section dealing with lubrication. The manufacturer rightly gives this section prominence and in it he insists on the use of certain oils and specifies the length of time for which they may be used. The car owner does not always regard this advice as seriously as he should and is apt to forget that it represents the combined experience of the motor manufacturer and of the oil industry.

These notes give a broad outline of the problems involved in lubricating the modern motor car so that the importance of proper lubrication may be seen in its true perspective.

Any piece of mechanism such as a motor car engine, in which relative movement takes place between two parts, requires lubrication. This implies the separation of two moving surfaces by a viscous fluid film. If this viscous film is not present and metal to metal contact takes place between the two moving parts, the resulting friction generates sufficient surface heat to cause local welding between the two parts. This is commonly called "seizure". When, however, the two parts are separated by a viscous fluid film then metal to metal contact and consequent seizure cannot occur.

The film can be regarded as being of three layers with the two outer layers adhering to the metal surfaces, and the centre layer, due to the relative movement between the outer surfaces, in a state of continuous fluid shear. In this way, irreparable disruption of the metal surfaces is replaced by continuously reparable disruption of a fluid. Provided that a true viscous film can be maintained between the two surfaces, the load which can be maintained between these surfaces without disruption of the lubricating film depends only on the viscosity of the film and not on its chemical or physical nature, a condition known as "fluid film lubrication".

If the load is sufficient to break down the film, an intermediate stage commonly called "boundary lubrication" applies before actual metal to metal contact takes place. When a metallic surface is wetted with a fluid, there is molecular attraction at the interfaces, i.e. between the metal surface and the immediate molecular layer of lubricant

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on that surface. The degree of adhesion depends on the physical and chemical natures of the metal surface and of the lubricating fluid. The stronger the adhesive force, the greater the load required to break this attracted or absorbed lubricating film away from the metal. The size of the molecules of the lubricant is of significance in these conditions of lubrication—the longer molecules usually requiring a greater load to remove them. The two lubricated surfaces of a lubricated bearing, therefore, can be regarded as being coated externally on the journal and internally on the bearing shell with a pile carpet, each individual hair of the pile representing a molecule of the lubricant. Between the two layers of carpet is a complete fluid film. As and when this film is broken down, actual metal to metal contact will take place at a load depending on the thickness of the pile of the carpet (i.e. the molecule size) and on the strength of the bond between the pile and the metal surface.

Thus the problem of properly lubricating the motor car engine, or any other piece of mechanism for that matter, can be reduced to the simple terms of maintaining a full fluid film of lubricant between all relatively moving parts under all load conditions or, where this may not be possible, increasing the break-down resistance under boundary layer conditions and, as a final resort, minimizing the effect of metal to metal contact, as and when it occurs, by chemical means.

ENGINE LUBRICATION

There is an optimum viscosity of lubricant for any bearing. If the viscosity is below the optimum, the film will break down and boundary conditions will come into being, whilst an increase in viscosity above the optimum produces an increase in fluid friction and, consequently, power loss in the bearing.

The motor car engine is a heat engine and operates under widely varying speeds, loads and temperatures so that it is impossible for the perfect lubricant to be supplied at all times to each bearing surface, but, as in all branches of engineering, a compromise must be reached.

Any fluid of adequate viscosity will provide proper lubrication, but experience has shown that the only fluid readily available in adequate quantities and able to meet the demands of the internal combustion engine is lubricating oil obtained from petroleum, which, according to the crude source from which it is obtained and the method of refining, can be produced in almost any desired viscosity.

Theoretically, it is desirable that an oil should be used having a viscosity sufficient properly to lubricate the most highly loaded bearing in the engine at its maximum temperature. It is a natural physical property of petroleum oils, however, that their viscosity should vary with temperature—the lower the temperature, the higher the viscosity. It is usual to select an oil having adequate viscosity to give proper lubrication under maximum temperature and load, which means that under all lower temperature and load conditions the oil is more viscous than it need be. For many years, the petroleum industry has been seeking to produce oils in which the viscosity change with temperature is reduced to a minimum. This is particularly important in so far as starting of the engine from cold is concerned. The higher the viscosity, the greater the power required to turn the engine over and, therefore, for a given battery and starter capacity, the cranking speed is reduced with increasing oil viscosity.

This effect of oil viscosity on cold starting can hardly be over-emphasized. Too low a cranking speed may mean that carburation is ineffective so that a proper starting mixture cannot be

obtained in the cylinders. It is also frequently noticed that in cold weather an engine will fire once or twice and then fail. This cycle is repeated until the sparking plugs become saturated with moisture of condensation, rendering a start quite impossible. Such a condition can be cured by a reduction in oil viscosity and is due to the fact that under the conditions of cold carburation, the power developed in the cylinder of the engine is insufficient to turn the engine over against the heavy drag of the viscous oil film, so that the engine can never start. In other words, the Friction Horse Power is greater than the Indicated Horse Power.

It has for some years been the practice to use what is known as oil dilution to facilitate the starting of aircraft engines in very cold weather. Before the engine is stopped, a valve is opened which introduces a considerable quantity of petrol, of the order of 15 per cent of the oil capacity, into the lubricating system, thereby diluting the oil and reducing its viscosity. When the next cold start is made, the diluted oil is of sufficiently low viscosity to enable adequate cranking speeds to be obtained and, as the engine warms up, the petrol is boiled off from the lubricating oil which, by the time full working temperatures are obtained, has been restored to its previous quality. Such deliberate dilution of the lubricating oil to facilitate cold starting cannot be used in the automobile engine, as the maximum temperature which is reached by the lubricant is never sufficient to boil off the diluent. The success of this practice makes it quite clear that dilution of the lubricating oil in a motor car by a small amount of petrol—contrary to widely accepted beliefs—will not have any deleterious effect. This does not mean, however, that the starting valve or strangler can be used indiscriminately under cold starting conditions because excess of petrol in the combustion chamber will cause misfiring, and a physical washing of the oil film from the cylinder walls, with the result that cylinder wear both due to corrosion and from piston-ring abrasion or flutter is accelerated.

It is in these cold starting conditions that an oil of low viscosity is beneficial apart from the way in which it facilitates the actual start. The lower the viscosity of the oil, the more easily it is pumped by the pressure lubricating system and the greater the quantity which will be flung on to the cylinder walls and other parts of the engine which are splash lubricated, thus reducing wear.

The writer remembers a test carried out one very cold morning on a car which had been left out all night. The sump was removed and an oil tray full of the normal service oil was held at the pump inlet. The engine was started and it was just over four minutes before any oil sling was seen at the big ends. During this time the pump discharge, which was reduced by cavitation, was going through the relief valve.

Having made a case for the use of oils which at low temperatures are sufficiently fluid to reduce oil drag and also give proper lubrication, their behaviour at high temperatures must next be considered.

The first requisite, as has been stated, is that the oil at the maximum temperature of any part of the engine should be sufficiently viscous to maintain full fluid film lubrication. Apart from this, it must be very resistant to oxidation. Petroleum oils, when subjected to heat in the presence of air, take up oxygen in varying degrees and the oxidized products are, in general, in the initial stages gummy or varnish-like in constitution and later become hard and coke-like. It is of the utmost importance, therefore, that the lubricating oil should be extremely resistant to oxidation so that the deposits of sludge and varnish-like material on the piston assembly and in various parts of the

crank-case may be reduced to the minimum. The black sludge which is frequently found in corners of the crank-case and in the valve chest of side-valve engines and in the rocker box of overhead valve engines, is not, in the main, an oxidation product of the oil. It is usually an emulsion of oil and water, the latter coming from condensation in the crank-case caused by blow-by and changes of temperature; the emulsion being made stable and black in colour by small particles of carbon. Modern technique leads towards controlling and reducing oxidation of the lubricating oil by the addition of small quantities of chemical oxidation inhibitors.

It is deterioration of the oil by oxidation which is the most important factor affecting the frequency with which oil changes have to be made. This assumes, of course, that solid material can be eliminated by proper filtration.

High-duty petrol engines, such as some aircraft engines, and high-duty diesel engines are prone to suffer from sticking of the piston rings and consequent mechanical troubles due in part to the oxidation of the oil and partly to oxidation of parts of the fuel which may not be completely burned. This latter applies particularly to the diesel engine. These effects can be controlled by the addition of what are called "detergent additives" to the oil. In one form the detergent additive is a metallic soap which has the same effect on the lubricating oil as toilet soap has in water. It prevents the deposition of the gummy deposits which cause ring sticking by holding them up in a colloidal suspension cum solution in the lubricating oil. The power output of the motor car engine in terms of cylinder size is much lower than that of the aircraft engine, and the power level at which it is operated is lower, with the result that sticking of piston rings from other than purely mechanical causes such as might follow a partial seizure is unknown. For this reason detergent oils are of less value in a conventional motor car, though they have virtue in reducing general crank-case deposits.

By proper design of the lubricating system all moving parts of the engine with the possible exception of the piston rings and, under certain conditions, the piston itself, can be maintained under full fluid film lubrication. There is still some doubt as to the precise conditions of lubrication under which the piston assembly operates, and under critical conditions such as are met in some aircraft engines, where piston ring scuffing may occur, the lubricant can be strengthened under boundary conditions by the addition of a small amount of an additive either of the fatty type or possibly a sulphur/phosphorus compound. Materials of this type adhere firmly to the metal surfaces and also are composed of long molecules so that what was described as the "pile carpet effect" in the introduction to this article is strengthened. Such additives must be used and selected with considerable care as there is otherwise a risk that they may tend to reduce the oxidation resistance of the oil.

In the writer's opinion, the lubrication system of the engine warrants far more attention in the design stage than it usually receives.

Although there are several brilliant exceptions, the majority of automobile engines have an inadequate oil capacity and the oil circulation rate is too low. The sump capacity of the average 12-h.p. engine should be at least 1½ gallons and the circulation rate 80 gallons an hour as a minimum for the family type of motor car and 120/150 gallons an hour for a high-duty engine. These figures should be increased to 2½ gallons sump capacity for a high-duty 4-litre engine with an oil circulation of the order of 250/300 gallons an hour. It is appreciated, of course, that the provision of adequate sump capacity is very difficult where forward mounted engines are concerned; clearance between

the front suspension and the engine being the limiting factor.

The one disadvantage of large sump capacities is the time required for a proper warm-up of the oil. This can, however, be materially reduced by suitable location of sump baffles and the oil pump inlet. A large sump capacity certainly reduces the risk of a temporary loss of oil pressure due to the oil surging away from the pump on a fast corner. Some cars with a capacity of less than one gallon are critical to within a pint in this respect.

Detailed design of oil pumps and feed galleries well repays proper care. It must be remembered that oil, particularly at high velocities, does not appreciate turning sharp corners, nor being subjected to sudden changes in pressure and velocity such as occur when changes of section are made in oil feed pipes and galleries. The entry to the oil pump could with advantage be increased considerably in size on most engines and should be equal in width to the gear centres, if a gear pump is used. The almost universal practice of driving the oil pump from the bottom of the distributor shaft is to be commended in that it ensures that a large slow-speed pump is used, which is always preferable to a small high-speed one of comparable capacity.

The writer is at the moment using a 12-h.p. motor car of a popular make and recent design on which the restriction on the inlet side of the pump is such that even with an SAE-20 oil, full-gauge pressure cannot be obtained in less than half an hour's running, due to cavitation. On the delivery side of the pump sudden changes in section and direction, although inevitable, should be reduced as far as possible. The oil ways in most engines could, with advantage, be larger. It costs, if anything, less to drill a long big hole than a long small one. The actual feed through the main bearing shells into the crankshaft system is another point warranting attention. The writer has vivid memories of his apprenticeship days and an experimental 10-h.p. car with a three-bearing crank-shaft on which the oil feed to the centre main bearing and, in consequence, to numbers 2 and 3 big ends, was through a 3/32-inch hole drilled in the dowel which located the centre main bearing shell. A simple arithmetical calculation in terms of cross-sectional areas of bearing clearances in relation to the feed way would have prevented the spate of bearing trouble to which this engine was prone until the oil feed was properly modified.

Much has been written on the subject of oil filters and discussions between the protagonists of full-flow filtration versus by-pass have not reached finality. The full-flow filter, in which the full delivery of the oil pump at all times passes through the filter, has much to recommend it especially on new engines where the elimination of foundry sand—the foreign material which cannot be excluded during manufacture—is most important. A by-pass filter will filter a smaller proportion of the oil rather more thoroughly than a full-flow of similar size, but provided there is adequate under-bonnet space, there seems no real reason why filters of both types should not be used—if necessary, incorporated in a common casing. There is an alternative system whereby one type of filter pack can be used to give full-flow filtration for the first 1,000 miles running, to be replaced at this period by a different type of pack giving by-pass filtration only, the main oil feed passing through the filter relief valve.

Filter design has, of recent years, improved out of all knowledge, not only in the actual filter medium but also in the detailed design of the filters. It is not many years since there were filters on the market which, when opened up for the removal of the filter element, discharged their entire contents, including the bulk of the trapped material,

into the oil system of the engine. The writer has also seen full-flow filters which were scoured of the trapped material every time the filter relief valve opened, an occurrence which took place every time a cold start was made.

On the suction side of the pump, if a filter is required at all, all that is needed is a coarse gauze to hold back material sufficiently large to damage the pump.

Oil cooling usually presents few problems. The heat dissipation capacity of the sump is nearly always sufficient to maintain the sump oil temperature at a reasonable and safe figure of the order of 70° C. though cars used extensively in the tropics may need additional cooling. An interesting system was incorporated on a 2·6-litre car introduced in 1939. The oil from the delivery side of the pump was taken into a coiled pipe contained in a gallery cast in the cylinder head and thence to the engine oil-distribution system. The advantages were twofold. The water in the cylinder head, due to thermostatic control of the water system, warmed up very rapidly, thereby warming the oil and quickly reducing its viscosity after an initial start. As soon, however, as the oil temperature exceeded the water temperature, the heat transfer was in the opposite direction—the cooling system tending to cool the lubricating oil.

The foregoing remarks have dealt with the distribution of lubricating oil to the crank-shaft and cam-shaft and similar moving parts. Lubrication of the piston assembly, including the gudgeon pin and small end, is usually by sling from the big ends. This is a variable factor and one that is dependent on bearing clearance and consequently on wear. The effects of wear on the big-end sling can be reduced by adopting the system which is coming into use on certain aircraft engines whereby the crank-shaft is fed with pressure oil from the ends only, so that the feed into each journal bearing is controlled only by the size of the feed hole in the crank journal and the pressure in the crank-shaft system. This obviates big-end starvation, due to excessive flow through the main bearings following wear. There is little that can be said about the piston assembly and control of oil consumption. The piston clearances are usually dictated by materials and temperatures and anticipated percentage of running time at full power, and the selection of oil control rings to keep the oil consumption within reasonable limits is a matter of trial and error. One American aircraft engine makes use of a novel piston-ring assembly. There are the usual gas rings at the top of the piston followed by a powerful scraper which is above the gudgeon pin. At the bottom of the piston skirt, below the gudgeon pin, is another scraper ring assembled "upside down". This is deliberate and the intention is to draw oil up over the piston skirt to ensure proper lubrication after which the oil is cut off by the powerful top scraper ring.

There are one or two other points on general engine design which have some bearing on lubrication.

On dry sump engines in particular, as fitted to some racing cars, much trouble can occur if the crank-case dimensions are kept too small, so that there is inadequate clearance between the crank-shaft assembly and the crank-case walls. An engine suffering from this trouble will not scavenge properly. Crank-case breathers could usually be increased considerably in size with much advantage to general internal cleanliness of the engine. This particularly applies to overhead-valve engines, which really require additional breather capacity on the rocker box. An increase in breather size might also persuade manufacturers to fit air filters on the breather. The spring-loaded relief valve which is usually fitted on the pressure side of the oil pump to avoid excessive pressures with cold oil is another part which well

repays careful design. It should be readily accessible for cleaning as a dirty relief valve is liable to stick or, at the best, to have a defective seating. The writer has little liking for the simple type of ball relief valve. The use of a properly designed poppet type of valve with 45 degree seating is well worth while. It is better to dispense with the relief valve altogether, and make the system capable of withstanding high oil pressures, than to fit an inadequate and poorly designed one.

TRANSMISSION LUBRICATION

In general there are fewer problems in regard to the lubrication of the automobile transmission than there are in the engine.

Until about 1935, there was a tendency to use very viscous oils both in gear boxes and in back axles, mainly to reduce noise. Latterly, however, an appreciation of the importance of accurate tooth profile and the necessity to provide proper support for the gears and their attendant bearings, and their effect in reducing noise, has enabled very much lower viscosity oils to be used with all the attendant advantages of reduced power loss and rapid establishment of lubrication from cold.

The introduction of the hypoid gear, and of some other types of spiral bevel, has increased gear-tooth loadings to a point where the ordinary viscous oil film is not sufficiently strong to prevent metal-to-metal contact and scuffing or seizure.

This problem has been tackled chemically and what are known as extreme pressure oils have been introduced. There is no complete unanimity of opinion as to the mechanism whereby such oils function but, in general, they can be divided into mild and active types—both the same in principle but differing in degree. The mild type usually contains a small proportion of a phosphorus or sulphur compound, which at the elevated local temperatures immediately following oil-film breakdown, and preceding local welding, reacts chemically with the surface of the gear teeth and forms an iron/phosphorus or iron/sulphur compound. This compound has a low melting-point and smears over the surface of the teeth rather like a french polish and prevents actual seizure. In a milder form, such phosphorus- or sulphur-containing oils can be used to accelerate running-in of a new transmission system or, in some cases, of a new engine. The high spots, which by the conventional running-in procedure have to be worn away gradually, are removed or smoothed over as phosphorus- or sulphur-containing compounds.

Where loads are very high, however, the mild type of E.P. oil may not be able to prevent seizure, and in these cases recourse is made to the active type of extreme-pressure oil which usually contains sulphur and chlorine. As in the mild type, these oils are comparatively inactive until high temperatures preceding seizure are reached. At these temperatures activity increases and sulphur- and chlorine-containing compounds are formed on the teeth surfaces, which are easily sloughed away from the surface of the metal without disrupting the base. Thus seizure is prevented, but at the expense of what may be called "chemical wear". The rate of this chemical wear, however, is so slow that it has no significant effect on the service life of the parts with which the oil comes into contact. Extreme-pressure oils, by their very nature, must steadily become deficient in active chemical constituents and therefore require replacing at definite intervals.

CHASSIS LUBRICATION

Two methods of chassis lubrication are in common use—the grease gun and the automatic oiling system. Both in fact use oil.

Grease is a mixture of lubricating

oil and soap, and physically can be regarded as a sponge of soap with the pores full of oil. Pressure on the sponge, as occurs when a grease-lubricated bearing is loaded, causes the oil to squeeze out so that it is available for lubrication. On release of the pressure the oil returns to the soap sponge.

The selection of the proper grease for each point requiring lubrication must be made with care. To generalize, the types of grease commonly available for automobile lubrication are manufactured from soda, lime and aluminium soaps which impart particular characteristics to the finished grease.

Soda soap greases will withstand high temperatures and are resistant to soap separation. They are therefore suitable for use in the ball bearings of electrical equipment. The soap is water soluble and these greases can not, therefore, be used for general chassis lubrication as road water will break them down.

The lime and aluminium soap greases by contrast are not water soluble, and therefore may be used as chassis lubricants. However, their melting-point is usually low, which makes them unsuitable for high temperature application in electrical equipment.

There are certain exceptions to the foregoing generalization, notably in water-pump greases which have to work at high temperatures in contact with water. Such greases are, however, usually too stiff for use in other than limited application. The importance of using the type of grease recommended by the maker should be clear.

Bearings which are grease lubricated require comparatively frequent attention as the clearance space between the journal and the bearing is the only reservoir. With the passage of time, however, there is a growing disinclination for the oil which is squeezed out to be taken back into the soap with the result that the bearings become dry and only soap is left. For this reason frequent attention with a grease gun is very necessary.

The automatic lubrication system, whereby oil from a central reservoir is piped through restrictors to the various chassis bearings, is, in the writer's opinion, very satisfactory. The oil can be pumped either automatically by means of a vacuum-operated pump worked from the engine induction system or by foot or hand operation. The writer has a preference for the latter, provided that it is possible to operate the system whilst the car is in motion, as then the oil can be fed to the chassis bearings with proper frequency. The disadvantage of the automatic system is that there is a tendency for the oil feeds to be restricted more than they should be to avoid complaints of oil spotting in the garage floor.

Oil-impregnated bearings of the "Oilite" type are also very satisfactory for certain applications.

The chassis lubrication system, whether it is by grease or oil, is always fighting the effects of water and road dirt and the manufacturer can do much to extend chassis life by the provision of really adequate seals on all chassis bearings, thereby at one and the same time preventing loss of oil from the bearing and ingress of dirt or water to it. Proper surface corrosion protective treatments on shackles, king pins and steering joints will prolong their life many times.

In conclusion, it should be emphasized that this note on the lubrication of the automobile is not intended to give more than a broad outline of the problems involved and to draw attention to the necessity for following professional advice and recommendations.

COUNCIL COMMENTARY

A VERY full Council assembled on February 12 for one of the usual monthly meetings and this time dealt with reports from Finance, Election, House and Publications Committees, together with miscellaneous items such as the Cadman Memorial Lecture, the next World Petroleum Congress, elections to Council and a report from Mr. Dewhurst on the Parliamentary and Scientific Committee.

There was also correspondence from the British Standards Institute and the Institute of Mining Engineers.

The House Committee has recently been strengthened by the co-option of Messrs. Dewhurst and Hyams and the report dealt with matters of accommodation and staff. A librarian-clerical assistant has already been appointed and taken up her duties, and considerable progress has been made towards ensuring our extended accommodation for the future.

Publications' staff has been hampered by the illness of Mr G. Sell, but is being helped over the difficulty by generous and competent assistance rendered by Mr J. D. Kelley, recently retired from the position of Editor of the A.I.O.C. journal, *The Naft*.

It is proposed to enlarge in this month's "Commentary" on the activities of the Branches Committee of Council.

This Committee is within sight of bringing to a conclusion a year of persistent work sponsored by the Chairman in connexion with the formulation of satisfactory Branch rules. It has always been recognized that healthy Branches in close and amicable touch with headquarters were essential to the full working of the Institute as a whole, and the past two years have seen a steady increase in representation of Branch members on Council, both in their own right and as *ex-officio* members by virtue of office held in a Branch. It would, in fact, hardly be claiming too much to say that this enhanced Branch activity is very largely responsible for the emergence of the *Review* as an additional publication and a particular link between Branches and headquarters.

The urge to get a set of new rules is by no means entirely, or chiefly, disciplinary, but rather for the purpose of assisting new Branches and co-ordinating the activities of existing Branches.

One of our stated objects is "to enable technologists to meet and correspond and to accumulate, extend, increase and disseminate information and knowledge about petroleum matters", and this can be done only very incompletely by meetings held exclusively in London. Even at a well-attended joint meeting with another Society in London we consider it good if we get an attendance of 150, and this scarcely covers a membership getting on for 2,000. The question of the constitution of Branches is, therefore, of considerable importance.

It has been recognized and embodied in the recently formulated rules that the Institute can do better work by gathering into the Branches non-corporate members rather than by being exclusive. Yet since the parent body carries ultimate responsibility for the action of Branches, it is necessary to have a majority of corporate members on Branch Committees. Furthermore, rules covering Branches must, by constitution, conform to the By-Laws of the Institute. For all these reasons patient and successful study has been given to drawing up a set of rules which it is hoped will soon be approved in final form.

It is recognized that in any given Branch there may be a predominance of members of a particular type, this predominance of one type varying from one Branch to another at home or abroad.

It is the endeavour of Branches Committee to maintain maximum flexibility within the limits of the By-Laws and, as a relatively new problem has been created by the post-war situation in connexion with the former London Students' Branch, the Committee is now dealing with concrete proposals for a modified type of Branch to meet these special conditions.

This will be fully discussed at Manchester at the next meeting of the Branches Committee.

These Branch Committees, incidentally, are held at Branch centres as well as in London—a good practice which ensures closer touch between Council and the Branches than could be maintained if all meetings were held in London.

PERSONAL NOTES

Mr J. B. Aug Kessler, a Fellow of the Institute of Petroleum and a Managing Director of the Shell Transport and Trading Co., Ltd., and other companies of the Shell Group, has been appointed General Managing Director of the associated Royal Dutch Company. Other changes reported on the Board of the Royal Dutch Company are the resignation as a Managing Director of J. C. van Panthaleon Baron van Eck and the appointment to succeed him of Jhr. Dr J. H. Loudon.

Mr Gilbert C. Whigham has resigned from the Board of the Anglo-Iranian Oil Co., Ltd., and Burmah Oil Co., Ltd. He is succeeded on the Anglo-Iranian Board by *Sir Kenneth B. Harper* (a fellow Director of the Burmah) and the vacancy on the latter company's Board has been filled by the appointment of *Sir Alexander S. Bilsland*. Mr Whigham, it is interesting to note, has been associated with the Burmah Oil Co. for the past forty-two years.

Mr H. D. Demoulins has been appointed a Director of the Anglo-American Oil Co., Ltd., and will be responsible for the refinery operations of the company and its subsidiaries.

Mr C. T. Brunner, a Fellow of the Institute of Petroleum and Acting Secretary of the Petroleum Board (to which he was appointed from Shell-Mex and B.P. Ltd.) has been invited to join the Ministry of Transport's Petrol Stations Technical Committee.

Mr C. F. Smith, who has been associated with Standard for the past thirty-five years, has been appointed a Vice-President of the Standard Oil Company (N.J.).

Mr L. B. Levi, a Director of Socony-Vacuum Oil Co., Inc., and Chairman of its Foreign Trade Committee, retired at the end of December after forty-eight years' service. He is succeeded by *Mr W. L. Faust*.

The death is reported at his home in California of *Mr Wilson Cross*, a former Chairman of the Vacuum Oil Co., Ltd., who retired in 1935.

Mr H. S. M. Burns has been appointed Vice-President of the Shell Oil Co., Inc., in the East of Rockies Territory. Since 1935 he has spent most of his time in Colombia in connexion with Shell's exploration and production work there.

Mr J. G. Pew has been appointed Vice-President in charge of production for the Sun Oil Company, succeeding his father, *Mr J. Edgar Pew*, who died in November.

Lieut.-General Sir Wilfred G. Lindsell, G.B.E., D.S.O., Ll.D., has joined the Board of Manchester Oil Refinery Ltd., and associated companies. During the last war he held such vitally important posts as Q.M.G. British Expeditionary Force (with responsibility for the landing of the B.E.F. and its maintenance in France) and, subsequently, Principal Administrative Officer G.H.Q., India, responsible for the development of the India Base and maintenance of the 14th Army and S.E.A.C. Forces in Burma.

The death occurred suddenly on February 10 of *Mr Walter Blumenthal*

Manager of Raven Oil Co., Ltd., the export organization of the Manchester Oil Refinery group of companies.

Mr F. C. Starling has retired from the post of Under-Secretary of the Petroleum Division. He is being succeeded by *Mr E. A. Berthoud* (formerly of the Anglo-Iranian Oil Co.) and *Mr V. Butler*.

Mr W. F. List has joined the Board of C. C. Wakefield and Co., Ltd., and will be responsible for overseas development. He spent over twenty years abroad for his company in India and South Africa and returned home in 1946 to become Overseas General Manager. *Mr L. M. Broadway*, Secretary of the company, has also been elected a Director.

Mr H. C. W. Johnston has resigned from the Board of Trinidad Leaseholds.

NEW ADDRESSES

The address of Petromar, Ltd. (Manchester Oil Refinery group) is now Jacey House, 16, Oxford Street, Manchester 1 (Tel. Central 6982).

Richard Klinger, Ltd., have opened a new branch office and depot at 11, Goree Piazzas, Liverpool 3 (Tel. Central 3497).

PETROLEUM EQUIPMENT

A recent illustrated bulletin issued by the National Supply Export Corporation (No. 312) gives details of the National (Adjustable) Micro-Chokes, Types D and E. The latter is chiefly applicable to high-pressure wells.

FORTHCOMING MEETINGS

INSTITUTE MEETINGS

Aromatics from Petroleum. H. Steiner. At 26, Portland Place, London, W.1. 5 p.m. April 9.

SCOTTISH BRANCH

Engines for Aviation—the Future. F. Rodwell Banks. At Royal Technical College, Glasgow. 7.30 p.m. March 28.

SOUTH WALES BRANCH

What Britain Has Done in the Oil Industry. A. E. Dunstan. At Britannic House, Llandarcy. 5.30 p.m. April 11.

STANLOW BRANCH

Fuels and Lubricants for Aero Gas Turbines. C. G. Williams. At Grosvenor Hotel, Ellesmere Port. 7.30 p.m. March 19.

NORTHERN BRANCH

Progress on the Synthesis of Fuels and Lubricants. Dr. Whalley. At Engineers' Club, Albert Square, Manchester. 6 p.m. March 18.

STUDENTS' SECTION (LONDON BRANCH)

Life in Oilfields To-day (Indian Punjab and the Middle East). E. S. Pinfold and E. N. Tiratsoo. At 26, Portland Place, London, W.1. 6 p.m. March 20.

MEETINGS OF OTHER SOCIETIES

The Application of Surface Chemistry to Colloidal Problems. Chemical Society (Tilden Lecture). At University College, Nottingham. 7 p.m. March 18.

Combustion Turbines. W. J. Calderwood Institute of Marine Engineers (Junior Lecture). At Croydon Polytechnic. 7 p.m. March 18.

Dryness of Steam and Priming in Marine Boilers. Com. (E) L. Baker, R.N., Institute of Marine Engineers, London. At Institute premises, 5.30 p.m. March 25.

Combustion and Combustion Equipment for Gas Turbines. Dr. E. A. Watson and Dr. Clarke. Institute of Fuel. At Institution of Mechanical Engineers, London. 6 p.m. April 9.

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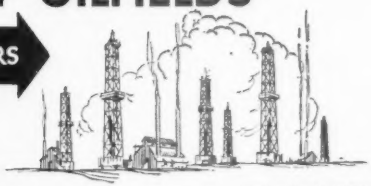

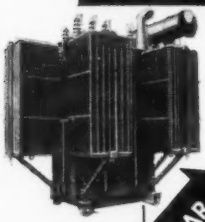

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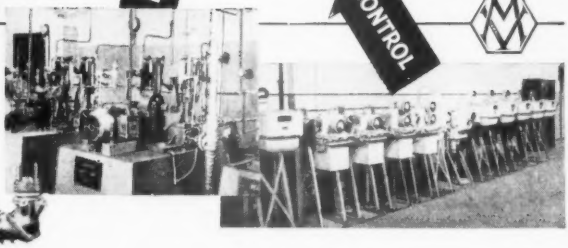

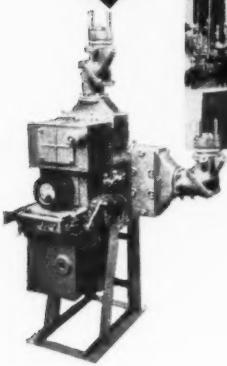

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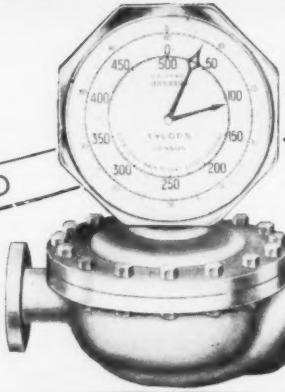

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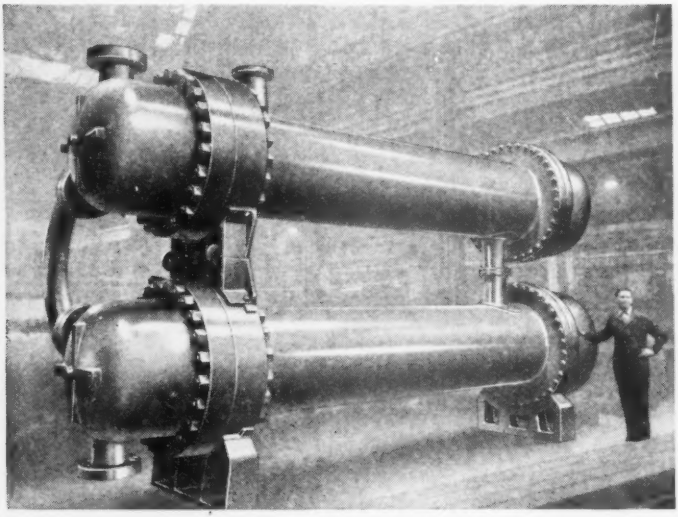

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